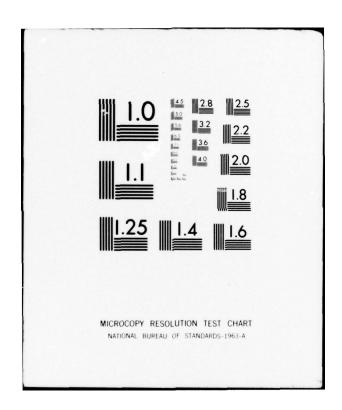
ARMY INST OF DENTAL RESEARCH WASHINGTON D C F/G 11/6 HIGH TEMPERATURE MICROSCOPY OF PORCELAIN-PRECIOUS ALLOYS, (U) AD-A039 647 APR 77 E F HUGET, L B DE SIMON UNCLASSIFIED NL OF AD A039647 END DATE FILMED 6-77



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HIGH TEMPERATURE MICROSCOPY OF PORCELAIN-PRECIOUS ALLOYS

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1 28 Apr 29

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Commercial materials and equipment are identified in this report to specify the experimental procedure. Such identification constitutes neither recommendation nor endorsement.

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of neighboring grains and slip. At 1,9500 F., all alloys showed pronounced grain distortion and incipient melting. Changes in surface architecture of the test alloys that occur at high temperatures may enhance the micromechanical interlocking of a fused veneer. However, such changes would appear to be detrimental to the fit of precision cast restorations.

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18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Dental alloys; high temperature microscopy; microstructure; porcelain veneer and porcelain-metal-restorations.

This study traced changes in the microstructures of five alloys over the range of temperatures employed in the application of dental porcelain. Specimens were 1/16 X 1/4-inch cast discs. A microscope that provided hot-stage and vacuum capabilities was used to monitor microstructures of the alloys between 800°F and 1,950°F. Heating rate of the cast pieces was 100°F per minute. Visualization of grain boundaries was made possible by selective thermal etching. Two alloys showed crystallographically dependent striated contours at temperatures in excess of 1,700°F. These markings appeared to result from nonuniform expansion

HIGH TEMPERATURE MICROSCOPY OF PORCELAIN-PRECIOUS ALLOYS

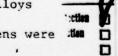
Throughout the years, microstructural studies have contributed to the characterization of dental alloys. Most observations on these materials have been made at room temperature. Therefore, attention has been directed toward structures which are stable at relatively low temperatures, or toward structures which can be obtained in the metastable condition by quenching from higher temperatures.

Alloys used for the fabrication of metal-ceramic restorations are subjected to repeated short-duration heat treatments during fusion of the esthetic veneer. Descriptions of microstructural alterations induced by such heat treatments have been reported. 1-4 However, these descriptions have been based upon room-temperature observations of structures revealed by chemical etchants. Heretofore, the dynamic microstructural features of veneerable high-fusing alloys have not been studied at elevated temperatures.

The present study was conducted to observe the structures of five alloys at the temperature employed in the application of dental porcelain.

MATERIALS AND METHODS

Test materials included four gold-palladium-silver based alloys $(A^*, B^+, C^{\S}, \text{ and } D^{\P})$ and a palladium-silver alloy (E^{Ω}) . Specimens were item



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 $1/16 \times 1/4$ -inch cast discs. The castings were fabricated by conventional lost wax laboratory procedures. Metallurgical papers (240-600 grit) and alumina abrasives (0.3 and 0.05 μ m) were used to polish the test face of each disc.

A metallurgical microscope that provided hot-stage and vacuum capabilities was used to monitor the microstructures of unetched polished castings between 800 and 1,950F. A schematic of the heating and monitoring devices is shown in Figure 1. Heating of the cast pieces at a rate of 100F per minute was accomplished with the use of a tungsten electric furnace. A vacuum of 10⁻⁵ to 10⁻⁶ torr was maintained within the heating chamber to retard oxidation of the specimens. Microstructures were observed at a magnification of X 400. The intercept (Heyn) procedure was used to determine ASTM micro-grain size numbers. 5

RESULTS

Microstructures of the five alloys were revealed by thermal etching. Grain boundaries, with the exception of those of alloy A, became visible at 1,100F. Alloy A specimens resisted thermal etching until the temperature of the heating chamber reached 1,500F. Grain-configurations are shown in Figure 2. Micro-grain size numbers for the test materials were: Alloy A, 6; alloy B, 7; alloy C, 5.5; alloy D, 7; and alloy E, 8. Exposure of the alloys to temperatures above those at which the grain boundaries became visible did not promote grain-growth.

H Unitron, Model BN-11, Unitron Instrument Co., Newton Highlands, MA.

Surface upheaval and changes in surface reflectivity accompanied further increases in treatment temperature. Alloy B was so affected at 1,500F. Changes in surface reflectivities of alloys A, C and E occurred at 1,700F. Alloy D exhibited a similar change at 1,900F (Fig 3). Additionally, alloys A, C and D showed crystallographically dependent striated contours at temperatures in excess of 1,700F (Fig 4). At 1,950F, all five alloys showed pronounced grain distortion and incipient melting.

DISCUSSION

The etching technique employed in this study differed from the more common metallurgical practices of "heat-etching" and "heat-tinting" in that the specimens were heated and observed in a vacuum. Hence, vaporization of constituent elements from relatively oxide free specimen-surfaces and observation of significant microstructural changes at elevated temperatures were made possible.

The preferential etching of grain boundaries at moderately high temperatures (1,100-1,500F) suggests that vaporization of irregularly arranged atoms at grain boundaries occurs at faster rates than vaporization of the more centrally positioned atoms. Decreased specimen reflectivity at somewhat higher temperatures may be linked to oxidation of base metal components such as indium or tin. Oxides of indium and tin play prominent roles in the chemical bonding of porcelain to metallic substrates. 6

Surface upheaval and intragranular striated markings displayed by three alloys appeared to result from nonuniform expansion of neighboring grains and slip. These changes in surface architecture may improve the micromechanical attachment of a ceramic veneer to a cast substructure. On the other hand, such changes would appear to be detrimental to the fit of precision cast restorations.

CONCLUSIONS

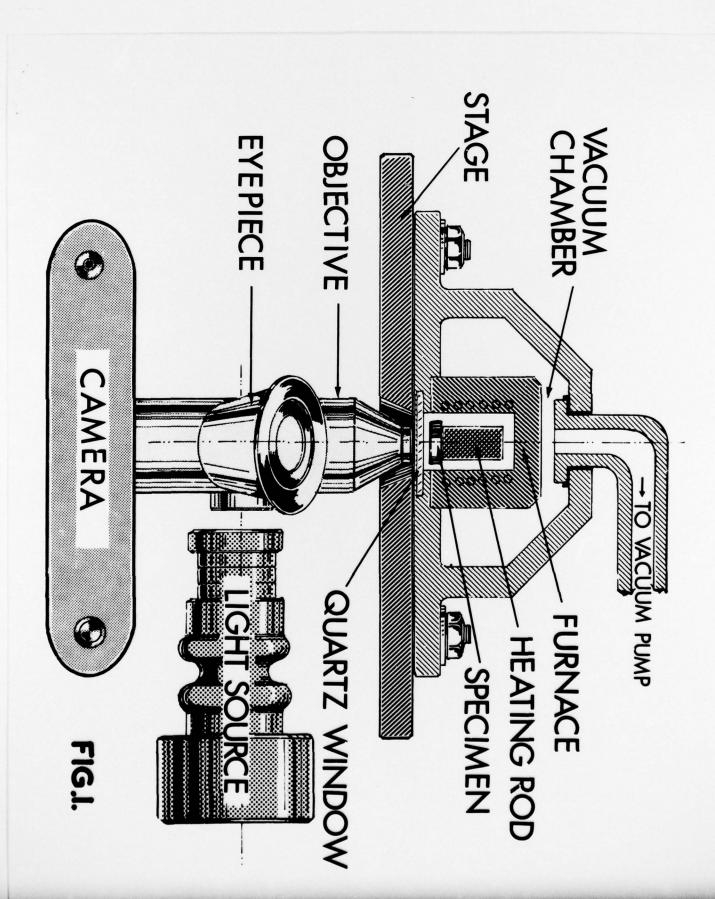
Five high-fusing dental alloys exhibited microstructural changes when heated at temperatures employed in the application of dental porcelain. Heating of specimens in a vacuum allowed observation of structural changes at elevated temperatures. The technique made possible the visualization of grain boundaries and twin boundaries, and the detection of changes in surface reflectivity and surface morphology. Hot-stage microscopy would appear to be useful for the study of all alloys exposed to high-temperature procedures during the fabrication of dental prosthetic devices.

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LEGENDS FOR FIGURES

- Figure 1. Specimen Heating and Monitoring Devices.
- Figure 2. Microstructures of Porcelain-Precious Alloys. (A) Alloy
 A, 1,500F. (B) Alloy B, 1,100F. (C) Alloy C, 1,100F.
 (D) Alloy D, 1,100F. (E) Alloy E, 1,100F.
- Figure 3. Microstructures of Porcelain-Precious Alloys. (A) Alloy
 A, 1,700F. (B) Alloy B, 1,500F. (C) Alloy C, 1,700F.
 (D) Alloy D, 1,900F. (E) Alloy E, 1,700F.
- Figure 4. Microstructures of Porcelain-Precious Alloys. (A) Alloy D, 1,800F. (B) Alloy A, 1,800F.



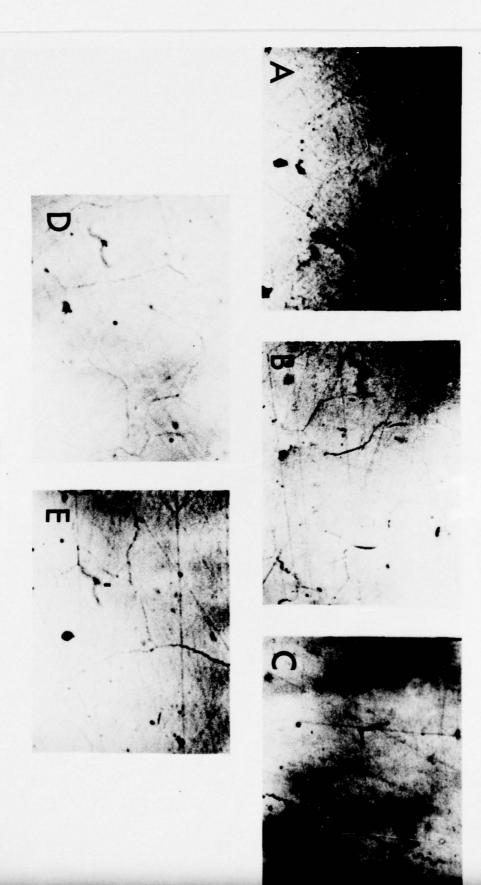


FIG.2.

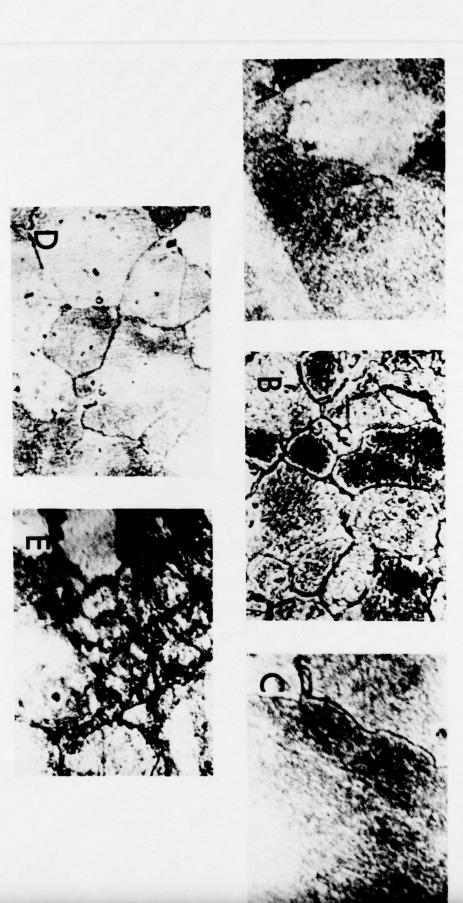


FIG.3.



FIG.4.